NATO Advanced Research Workshop

Broadband Dielectric Spectroscopy and its Advanced Technological Applications

Book of Abstracts

Perpignan, France
21 - 24 September 2011
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Scientific programme

Thursday, September 22nd, 2011

8:45 Opening ceremony

Rotational and translational diffusion as measured by BDS (Experiment)  
(Chair F. Kremer)
9:00 Yuri Feldman, "Dielectric spectra broadening as the signature of dipole-
matrix interaction; Water in complex systems"
9:30 Roland Böhmer, "Relaxation in supercooled alcohols"
10:00 A. Sokolov, “Decoupling phenomena in dynamics of soft materials”

10:30 Coffee break

11:00 R. Nozaki, "High-frequency dielectric response of alcohols between 100
MHz and 2.5 THz"
11:30 F. Ladieu, "Nonlinear susceptibilities of a supercooled liquid close to the
glass transition"
12:00 M. Wübbenhorst, "About compensation law, Meyer-Neldel rule and
"unphysical" activation parameters"

12:30 Lunch

Rotational and translational diffusion as measured by BDS (Experiment)  
(Chair Yu. Feldman)
14:30 J. Colmenero, "Broadband dielectric spectroscopy: a selective tool for
unveiling segmental and chain dynamics in asymmetric polymer blends"
15:00 V. Raicu, "A unitary approach to relaxation and dispersion in fractal
networks: From dielectric and mechanical relaxation to distribution of
income"
15:30 A. Serghei, "Broadband dielectric spectroscopy: from bulk to attograms of
matter"

16:00 Coffee Break

16:30 P. Ben Ishai, "Anomalous diffusion, Cole-Cole relaxations and the space
in which they occur: Puzzles and problems"
17:00 Ralph H. Colby, "Electrode Polarization and Dielectric Constant of Single-
Ion Conducting Ionomers"

17:30 Poster Session I
Scientific programme

Friday, September 23rd, 2011

Rotational and translational diffusion as measured by BDS (Theory, Chair V. Raicu)
9:00 R. Hilfer, "Applications and implications of fractional dynamics for dielectric relaxation"
9:30 W. T. Coffey, "Anomalous dielectric relaxation"
10:00 R. Metzler, "Time averages and breaking of ergodicity in complex systems"
10:30 Coffee break
11:00 R. R. Nigmatullin, I. I. Popov, E. Yu. Koroleva, A. A. Nabereznov, "The generalized Jonscher's law for conductivity and its realization for porous structures"
11:30 Yu. Kalmykov and S. V. Titov, "Characteristic times of anomalous relaxation processes"

12:00 – 14:00 Lunch

Rotational and translational diffusion as measured by BDS (Theory)
(Chair R. Metzler)
14:00 A. Schoenhals, "Molecular dynamics in nanometer confinement"
14:30 L. Trakhtenberg, "Dielectric properties of multifunctional metal-containing nanocomposites: non-phenomenological description"
15:00 M. Sega, "Simulating dielectric spectra for electrolytes and ionic liquids"

15:30 Coffee break

15:50 Panel Discussion

17:30 Poster Session II

19:00 Conference Dinner
Scientific programme

Saturday, September 24th, 2011
Molecular dynamics in nano-metric confinement (Chair P. Ben Ishai)

9:00 F. Kremer, "Dielectric relaxation at nano-metric length scale"
9:30 G. Floudas, "Heterogeneous polymer dynamics near Tg by single molecule spectroscopy"

10:00 Coffee break

10:00 S. Napolitano, "The lifetime of the deviations from bulk behavior in polymers confined at the nanoscale"
11:30 J. Sangoro, "Charge transport in confined ionic liquids"

12:00 Final conclusions
Abstracts
Dielectric spectra broadening as the signature of dipole-matrix interaction; Water in complex systems

Yuri Feldman, Alexander Puzenko, Paul Ben Ishai, and Eugenia Levy

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Whenever water interacts with another dipolar or charged entity, there exists a broadening of its dielectric relaxation peak. This broadening can be described by the phenomenological Cole-Cole law (CC), which can be represented by a frequency dependent complex dielectric permittivity \( \varepsilon^*(\omega) \):

\[
\varepsilon(\omega) - \varepsilon_\infty = \frac{\Delta \varepsilon}{1 + i\omega \tau}^\alpha
\]

(1)

where \( \omega \) is the cyclic frequency, \( i \) is the imaginary unit, \( \varepsilon_\infty \) is the high frequency limit of the complex dielectric permittivity, \( \Delta \varepsilon \) is the dielectric strength of the relaxation process, \( \tau \) is a characteristic time scale and the exponent \( \alpha \) is referred to as a measure of symmetrical broadening in the dielectric losses relaxation peak (0<\( \alpha \)≤1). A new phenomenological approach has been recently presented [1] that clarifies a physical mechanism of the dipole-matrix interaction in complex systems (CS) underlying the CC behaviour.

The model definition of the specific macroscopic dipole moment, when combined with the dependences of \( \alpha \) and \( \tau \) versus any external variables, will allow the development of the theoretical set of 3D trajectories for different systems (See Figure 1). Our hypothesis is that these trajectories will help elucidate one of the main challenges in dielectric physics today. Namely, to uncover a physical model underlying the CC behavior in complex systems, including biological materials.

![Figure 1. 3D Trajectories of CC relaxation processes: a) for water confined in the of the porous silica glasses; b) aqueous solutions of KCl at 25 C.](image)

The talk will demonstrate how the model described above can define the state of water in complex systems, including the biological ones. The model sheds light on the exchange between the bound and bulk water around a biological system by concurrently studying the dynamics and structural aspects of the relaxation peak.

Relaxation in supercooled alcohols

Catalin Gainaru, Sebastian Schildmann, and Roland Böhmer

Fakultät für Physik, Technische Universität Dortmund, 44221 Dortmund, Germany

Debye devised his 1913 relaxation model with the dielectric properties of water and alcohols in mind [1]. These liquids are subject to intense research because they are of fundamental importance for biophysical processes and, e.g., for the microwave ovens we use every day [2]. Nevertheless, the way to the microscopic understanding of these liquids has been beset with apparently conflicting observations and conceptual difficulties. While much of this remains true for water, the situation for alcohols is different. In this presentation, recent results are reported and discussed in order to demonstrate that a coherent understanding of these substances is emerging [3-5].

Fig. 1: Broadband dielectric loss spectra of (BuOH)_{0.78}(BuBr)_{0.22} [4].

Decoupling phenomena in dynamics of soft materials

Alexei P. Sokolov

Department of Chemistry, University of Tennessee Knoxville, Knoxville, TN 37996-1600, USA; and ORNL, Oak Ridge, TN 37831-6197, USA

Classical theories predict that structural relaxation of the liquid controls diffusion of molecules and ions in the liquid. However, there are many examples where this simple picture breaks down: (i) Self-diffusion is decoupled from structural relaxation (varies with temperature much slower than the structural relaxation time $\tau_\alpha$) in small molecular liquids; (ii) Chain modes are decoupled from segmental relaxation in polymers; (iii) Ionic conductivity decouples from structural relaxation in glass-forming systems, etc. The mechanism of this decoupling remains unclear.

We start the presentation from an overview of the decoupling between self-diffusion and $\tau_\alpha$ in small molecular systems and between chain and segmental modes in polymers. We demonstrate that the decoupling phenomena in both classes of materials have similar property and increases with increase in fragility of the system [1]. We relate the decoupling to dynamic heterogeneity. The observed similarity is explained by the diffusive-like nature of the chain modes in polymers [1].

Next we focus on decoupling of ionic conductivity from structural relaxation in ionic liquids and polymers. Our analysis [2] reveals surprisingly weak decoupling in room temperature ionic liquid. It is significantly weaker than expected one for so fragile liquids. In contrast, we found very strong decoupling of ionic conductivity in polymers, and decoupling in this case increases with fragility of the polymer [3]. We discuss possible microscopic mechanisms of this strong decoupling and its potential for design of solid polymer electrolytes for battery applications.

Broadband Dielectric Spectroscopy and its Advanced Technological Applications

Dielectric response of alcohols between 10 MHz and 2.5 THz

Y. Yomogida, a T. Iwasaki, b M. Matsumoto, a T. Mishina, b and R. Nozaki a

a Laboratory of Dielectrics and b Laboratory of Optical Properties and Semiconductor Physics, Department of Physics, Faculty of Science, Hokkaido University, Sapporo 060-0810, Japan

Dielectric response of hydrogen-bonding liquids is quite different from that of usual polar liquids. Water and some alcohols with one or two hydroxyl groups (OH) show a Debye-type dielectric relaxation with large relaxation strength, so called the primary process, and the secondary process with small strength in the high-frequency side of the primary process. This feature is very common for many alcohols even with different chemical structure. However, origins of these processes are still under discussion. Recently, terahertz (THz) dielectric spectroscopy has made it possible to observe the complex permittivity of alcohols in the frequency range up to several THz and to discuss on these processes in detail together with higher frequency processes [1-4]. Figure 1 shows dielectric loss behavior of 1-propanol between 10MHz and 2.5THz [1]. We have indicated that the experimental loss data is well described by using three Debye-type relaxation processes (1, 2 and 3) reported previously [5] and two damped harmonic oscillators (4 and 5). We will considere origin of these processes by changing the chemical structure of alcohols. We are also going to discuss difference and similarity in the dielectric relaxation processes of alcohols, fatty acids, methyl ketons and others to understand the origons.

Nonlinear susceptibility experiments in a supercooled liquid: evidence of growing spatial correlations close to $T_g$.


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The possible existence of a growing correlation length associated with the strong increase of relaxation times of glass-formers close to the glass transition is still a major open question in glass physics. It has been recently proposed by J.-P. Bouchaud and G. Biroli [1] that the ac nonlinear susceptibility of a supercooled liquid close to the glass transition temperature $T_g$ is a probe of dynamical correlations, and gives the temperature dependence of the number $N_{corr}$ of dynamically correlated molecules. As for spin glasses, where the nonlinear susceptibility diverges at the transition, this quantity is tailored to reveal the possible “hidden” critical behavior of the glass transition. We have developed a high sensitivity experiment to measure the nonlinear dielectric susceptibility of an insulating liquid at finite frequency, by measuring the third harmonic of the current flowing out of a capacitor with the supercooled liquid as the dielectric layer. This experiment is based on a bridge with two capacitors, and reaches a sensitivity better than $10^{-7}$ (ratio of third to first harmonics)[2].

Our first results on glycerol [3] will be presented and discussed. We shall show that, for a given temperature $T$, the modulus of the nonlinear susceptibility has a humped shape and reaches its maximum for a frequency close to the relaxation frequency of the liquid at that temperature. Moreover the magnitude of this hump increases as $T$ decreases towards $T_g$: this reveals directly the growing of $N_{corr}$ when approaching the glass transition. Our results thus reinforce the picture of an underlying critical nature of the glass transition. The data are consistent with the existence of two contributions to the nonlinear dielectric susceptibility: The first one is related to the correlations of interest here, while the second one is due to the saturation of the polarization[4].

About compensation law, Meyer-Neldel rule and "unphysical" activation parameters

Michael Wübbenhorst

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Many thermally activated phenomena such as diffusion in bulk solids, electronic conduction in amorphous semiconductors and (plastic) crystals, dielectric relaxations and thermally stimulated processes in polymers obey the compensation law or Meyer-Neldel (MNR) rule [1]. This phenomenon comprises a correlation between the apparent activation energy and the pre-factor in the Arrhenius law. Typical manifestations of the MNR are pre-factors for relaxation rates far above the phonon frequency ($f_0$ up to $10^{40}$ s$^{-1}$) or charge hopping rates in the order of $10^{28}$ s$^{-1}$.

This presentation gives an overview about the phenomenology of MNR behavior as well as some microscopic models for this phenomenon. Some specific cases will be discussed in detail that involve relaxations in plastic crystals[2], ionic conductors and glass forming systems below and above $T_g$. Though all examples show a similar signature implying a common physical basis for the occurrence of the compensation behavior, we have to involve various individual physical mechanisms to give a reasonable microscopic explanation.

Finally we will discuss arguments based on cooperativity, multiple excitations [3] or, generally, multi-body interactions in search for a common interpretation schema for the MNR. In this context the classification schema into non-cooperative and cooperative motions, introduced by Starkweather [4] and based on the Eyring equation, will be described and evaluated.

Broadband dielectric spectroscopy: a selective tool for unveiling segmental and chain dynamics in asymmetric polymer blends

J. Colmenero

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University of the Basque Country, San Sebastián, Spain

Miscible polymer blends are systems of wide technological interest whose dynamical and rheological properties can be tuned by varying the mixture composition. They are also very interesting systems from a basic scientific point of view. Even being miscible, these systems are dynamically heterogeneous: starting from two homopolymers with different mobility (different glass transition temperatures $T_g$) two separated segmental (a) relaxations are still observed in the miscible blend state. When the difference in segmental mobility ($T_g$) of the two-homopolymer components is not too high, they exhibit qualitative similar dynamic features in the blend. However, a rather different scenario emerges for low concentration of the fast component, if the two homopolymers display very different $T_g$'s. In this case, the two components in the blend exhibit strong dynamic immiscibility, with a large separation (dynamic asymmetry) in their respective a-relaxation times. In such conditions the motion of the fast component seems to be strongly restricted (“confined”) by the slowly relaxing matrix formed by the slow component. Recently, we have extended the investigations of dynamic properties of asymmetric polymer blends to the large-scale dynamics (chain dynamics) mainly by dielectric spectroscopy [1] and molecular dynamics simulations [2]. We found that the anomalous dynamic features induced by the slow matrix on the fast component also extend to this regime. The dynamic asymmetry induces strong non-exponentiality of the Rouse modes for the fast component. Moreover, we observe a striking crossover, by increasing the dynamic asymmetry in the blend, to a regime resembling scaling features characteristic of entangled-like chain dynamics, despite the used chain length being much shorter than the critical value for entanglement. We associate these features to strong memory effects that are induced by the slow nature of the confining matrix and thereby implying strong correlations between the external forces acting on the tagged chain. Theoretical methods based on generalized Langevin equations seem to be the suitable framework to describe chain dynamics in asymmetric polymer blends.

A unitary approach to relaxation and dispersion in fractal networks: From dielectric and mechanical relaxation to distribution of income

Valerică Raicu\textsuperscript{a,b} and Michael Stoneman\textsuperscript{a}

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\textsuperscript{b}Department of Biological Sciences, University of Wisconsin-Milwaukee, Wisconsin, USA

"Complex systems" is a generic term that is used across several scientific disciplines and includes: correlated systems of dipoles and spins in physics, protein networks, genetic networks and food webs in biological sciences, chemical reactions in inhomogeneous media, social and economic networks, etc. The study of relaxation is an active area of research in the fields of dielectric and nonlinear optical spectroscopy \cite{1}, which is insufficiently developed for the case of complex systems. It is currently firmly established that the relaxation of correlated systems of dipoles deviates markedly from classical Debye dispersion function (in the frequency domain) or from pure exponential decay (in the time domain) \cite{2}, but the exact ways in which these deviations occur and their significance are still debated issues. Here we propose that a fractal-tree network appropriately describes the energy relaxation pathway in virtually any complex system, and incorporates coupled (or hierarchical) as well as uncoupled (parallel) relaxation processes. This approach has been originally introduced for physical description of dipolar systems with memory, i.e., systems that present hierarchical (fractal) relationships between their parts \cite{3,4}. This talk will show that, upon adequate generalization and re-interpretation, the concept of relaxation sheds new light on processes taking place in complex systems, including dielectric relaxation in tissues, mechanical relaxation in proteins, actor collaboration networks, and distribution of income in populations of humans, to name but a few of the problems that can now be tackled with this approach.

\cite{1} V. Raicu, M. Schmidt, M. R. Stoneman, \textit{Structural dynamics and kinetics of myoglobin-CO binding: time-resolved X-ray diffraction and four-wave mixing}, In: V. Yakovlev (Editor), \textit{Biochemical Applications of Nonlinear Optical Spectroscopy}, CRC (2009).
Broadband dielectric spectroscopy: from bulk to attograms of matter

A. Serghei

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Investigations on attograms of material (1 attogram = 10^{-18} grams) open the door for fundamental questions in soft-matter physics, such as for instance “What is the minimum amount of matter necessary to “define” the material properties?” and – in the same time – offer new routes to be explored for enhancing the performance of macromolecular materials. The present contributions aims to demonstrate the strength of Broadband Dielectric Spectroscopy in measuring extremely small amounts of material. It will be shown that dielectric measurements on attograms of matter are possible by using a novel experimental concept which employs nano-containers as measurement cells (Fig. 1).

![Dielectric Spectroscopy Diagram](image)

Fig. 1: Nanocontainers as experimental cells to hold and measure attograms of material. The nanocontainers are identical, independent and additive. (a) schematic representation of the sample cell (b) SEM image of the nanocontainers in cross-section; (c) dielectric measurements on cooling and heating showing the phase transitions (crystallization, melting, Curie-transitions) for 80 attograms of PVDF-TrFE.
Anomalous diffusion, Cole-Cole relaxations and the space in which they occur: Puzzles and problems.

Paul Ben Ishai, Alexander Puzenko, and Yuri Feldman

Department of Applied Physics, The Hebrew University of Jerusalem, Givat Ram, Jerusalem

The Cole-Cole equation appears phenomenologically to describe dielectric relaxations in many, widely disparate systems. The meaning of this apparent universality is still a hotly debated topic. One aspect of this debate is the significance of the broadening parameter, $\alpha$. One may derive the Cole-Cole (CC) expression from a fractal Fokker – Plank equation[1]. In this case alpha expresses the fractality of the time set describing the interaction of our test dipole with its surroundings[2], [3]. Thus CC is linked to a continuous time random walker process (CTRW). How valid is the CTRW description as an expression of Universality? Having stated thus, an immediate consequence of this interpretation is the mean square path relationship $R^2 \propto t^\beta$. One must ask if the mesoscale structure of our systems is reflected in this approach.

On the other hand space scaling is somehow intimately related to another aspect of the CC equation, namely the dielectric strength, $\Delta \varepsilon$, via its implicit dependence on the dipole moment and correlation radii, another relatively unexplored avenue. What then is the relationship between these different length scales? How does the microscopic picture describing the evolution of a correlation function in the presence of local forces relate to the macroscopic dielectric strength?

Our paper does not intend to provide the answers to such profound questions. Rather we will demonstrate some possibilities that we hope will stimulate an exploration into this fundamental element of CC dielectric relaxation.

Electrode polarization and dielectric constant of single-ion conducting ionomers

Ralph H. Colby, Reuben Bushnell, and U Hyeok Choi

Materials Science and Engineering
Pennsylvania State University
University Park, PA 16803 USA

Polarization of ions at electrodes can be exploited to make devices that convert electrical energy to mechanical energy: actuators [1]. We explore the effects of electrode surface area and applied voltage on the charge polarization at the electrodes (EP), using a.c. and d.c. dielectric methods. At low applied voltages, the 1953 Macdonald model [2] (a mean-field solution to the Poisson-Boltzmann equation) describes the monotonic decay of the concentrations of ions polarizing near each electrode. Despite the fact that there is a large relaxation involving ions exchanging states just before EP, making the dielectric constant frequency-dependent, we utilize the Macdonald model to characterize the static dielectric constant of single-ion conducting ionomers and the number density of simultaneous charge carriers and their mobility. Such analysis is strongly dependent on the surface area and structure of the electrodes. At higher voltages (still below any electrochemical limit) the 2007 Kornyshev model [3] is used to understand the multi-layer structure of positive and negative ions near each electrode and why the efficiency of ionic actuators plummets as voltage is increased. Actuators require large numbers of simultaneously conducting ions and electrodes with enormous surface area, suggesting new design considerations [4].

Applications and implications of fractional dynamics for dielectric relaxation

R. Hilfer

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Broadband dielectric spectroscopy of glass forming liquids allows to investigate dielectric relaxation processes over almost 20 decades in time or frequency. Many materials such as 5-methyl-2-hexanol, glycerol, propylen carbonate or methyl-m-toluate exhibit a so called excess wing at high frequencies. Theoretical derivations for these experimental observations as well as simple fitting expressions are missing, and the observations are usually fitted with two Havriliak-Negami functions [1]. The presentation will discuss simpler fitting functions with fewer parameters [2,3,4]. These fitting functions are derived from the emergent theory of fractional time [2,5]. The theory of fractional time is based on the temporal coarse graining of translation groups or semigroups [2]. It has found numerous applications, especially in continuous time random walk models for anomalous transport [6], because of a general and rigorous mathematical equivalence [7]. Fractional dynamics is shown to have not only interesting theoretical implications for the concept of time evolutions, but also useful applications to experiment.

Anomalous dielectric relaxation in the context of the Debye model of noninertial rotational diffusion revisited

William T. Coffey, Yuri P. Kalmykov, and Serguey V. Titov

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The Debye theory of dielectric relaxation of an assembly of polar molecules as reformulated by Coffey et al. [1] in terms of a fractional noninertial Fokker-Planck equation for the purpose of extending that theory to explain the Cole-Cole anomalous relaxation is revisited in view of recent criticisms summarized by Henry et al. [2] of the use of that equation when time dependent external potentials are involved due to the non-commutability of the differential operators. It is shown that the theory proposed by Coffey et al. is consistent, however, with the ad hoc generalization for time dependent jumps of the Montroll-Weiss CTRW formalism proposed by Henry et al. [2] while the modified fractional Fokker-Planck equation, taking account of the non-commutability, fails to account for the form of the experimentally observed Cole-Cole spectra.

(Non)-Ergodicity and ageing in complex systems

Ralf Metzler

Physics Department, Technical University of Munich, Germany
Physics Department, Tampere University of Technology, Finland

Anomalous diffusion is a quite ubiquitous phenomenon in a broad range of complex systems, dominating the dynamics in systems ranging from the semiclassical realm, for instance, the dynamics of quantum dot blinking and laser cooling, over mesosopic systems such as diffusion in biological cells or charge carrier motion in amorphous semiconductors, up to macroscopic scales in groundwater tracer dispersion [1]. Often, the stochastic anomalies effect non-ergodic signatures in the behaviour of the system under consideration, for instance, in quantum dot blinking [2] or in the diffusive motion of lipid granules in living cells [3]. In this talk I will introduce the basic signatures of non-ergodicity and ageing by comparison of time versus ensemble averaged quantities [4,5]. In particular, subdiffusive continuous time random walk processes, often used in the modelling of dielectric phenomena, are non-ergodic due to the diverging characteristic waiting time scale. Fractional Brownian motion, another important mechanism describing subdiffusive behaviour, in contrast, is ergodic. However, it relaxes algebraically slowly to equilibrium. In both cases typical process characteristics are discussed, such as ageing plots, the behaviour of moments, and the velocity autocorrelation. Moreover, I will address the questions of linear systems response, and fluctuations of the dynamic patterns, giving rise to inhomogenous behaviour in ensembles.

The generalized Jonscher's law for conductivity and its realization for porous structures

R. R. Nigmatullin, a I. I. Popov, a E. Yu. Koroleva, b,c and A. A. Nabereznov b,c

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The new theoretical expression related to the Jonscher's correction for the complex conductivity that determines the contribution of carriers in frequency region has been derived. It has been shown that the conventional Jonscher's term in conductivity represents itself the partial case of more general expression, which is determined by ourselves as the Jonscher's generalized law for conductivity for a wide class of disordered media

\[ \sigma(\omega) = \sigma_0 + \frac{\chi^e\tau^{-\nu}}{1+(i\omega\tau)^{-\nu}} \]

In the case of strong fields when the characteristic relaxation time \( \tau \to 0 \) the suggested expression is reduced to the conventional Jonscher's expression, i.e. \( \lim_{\tau \to 0} \sigma(\omega) = \sigma_0 + \chi^e(i\omega)^\nu \). The basic idea that was used for the derivation of new law is based on the supposition that the disordered medium considered has self-similar (fractal) property [1, 2]. The proposed law is confirmed experimentally on dielectric data obtained for sodium nitrite imbedded in porous glasses. The justified and additional evidences helping in identification of this law (that can compete with other similar hypothesis) were found also. It has been shown that parameters entering into new expression are tightly related with other parameters describing the relaxation peak. So, based on new expression derived there is a possibility to extract additional information about relaxation behavior of dipoles from data related to their conductivity behavior. This interesting feature opens new information possibilities at analysis of relaxation peaks in cases when the contribution of relaxation processes into general expression for the complex dielectric permeability is small and is almost unnoticeable on the background of considerable contribution of conductivity processes.

Characteristic times of anomalous diffusion in a potential

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Despite the similarity in the solutions of normal and fractional diffusion equations, an essential difference appears insofar as the characteristic times associated with normal diffusion such as mean first passage time, integral relaxation time, etc. do not exist in anomalous diffusion (which is a natural consequence of the underlying Lévy distribution) [1]. The divergence of the characteristic times for anomalous diffusion, as defined in their conventional sense rendering them useless as a measure of the relaxation behavior, signifies the importance of seeking characteristic times for such processes in terms of their frequency domain representation. Here we shall show that the anomalous diffusion process in a potential will have several associated characteristic times. These can be expressed in terms of the fractional exponent and characteristic relaxation times of the normal diffusion process which appear as parameters in the definitions of the characteristic times of the anomalous diffusion process [2]. Thus, the difficulties associated with divergent characteristic times in anomalous diffusion may be avoided. Knowledge of the characteristic relaxation times is sufficient to accurately predict the anomalous relaxation behavior for all time scales of interest. As a particular example, we consider the subdiffusion of a particle in a bistable potential [2,3].

Molecular dynamics in nanometer confinement

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During recent years the investigation and understanding of the behavior of molecules in nanoscale confinements and close to surfaces attracts a growing interest. From the point of basic research the influence of finite size effects on the structure and dynamic of matter can be investigated experimentally and compared with theoretical approaches. On the other side the confining of molecules to nanopores or thin films is of importance for many practical applications for instance in chemistry and medicine.

Dielectric spectroscopy in the frequency range from $10^{-2}$ Hz to $10^9$ Hz is employed to study different systems:
1. The unusual relaxation behavior of water molecules confined to nanopores and to layered structures [1-4].
2. Molecular dynamics of liquid crystals confined to the nanopores of molecular sieves with different pore sizes [5-8].
3. Molecular dynamics of ultra-thin films [9,10].

In several cases the results obtained by dielectric spectroscopy are compared with data from broadband specific heat spectroscopy and inelastic/quasielastic neutron scattering.


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Dielectric properties of multifunctional metal-containing nanocomposites: non-phenomenological description

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The different types of two component nanocomposites have been investigated. These nanocomposites are vapor deposited metal-polymer films, porous glasses with metal nanoparticles, and metal oxide films containing SnO$_2$ and In$_2$O$_3$ nanoparticles. Dielectric properties of the materials were investigated using Broad Band Dielectric Spectrometer (NOVOCONTROL BDS 80) in wide frequency (0.1 Hz - 1 MHz) and temperature (−100 °C - 300 °C) ranges. The dielectric behavior of such composite materials confirmed the existence of different electron hopping mechanisms in the studied nanocomposites. For description of dielectric response of the metal/semiconductor containing nanocomposites a non-phenomenological model has been proposed based on hopping of electrons between traps around the metal nanoparticles [1].

At addition of the metal nanoparticles in amorphous matrix-isolator (glasses, polymers) the quality changes occur in the complex function of dielectric permittivity $\varepsilon(\omega, T)$ of the system. The theory describing these changes is developed here. This theory bases on the assumption, that in amorphous material takes place the great concentration of electron traps with the essential binding energy. These traps could be partially occupied by the electrons near nanoparticles. The trapped electron with the neighbor nanoparticle represents the large dipole moment. The turning of this moment in the external electric field occurs due to the electron jumps over the sphere near the surface of nanoparticle. The dielectric permittivity of the matrix is taking into account at the calculation of the interaction of a dipole moment with an external field. At first the dipole relaxation function $\{\Phi(T; t)\}$ of the system is calculated. Then the real and imaginary parts of $\varepsilon(\omega, T)$ are derived using $\Phi(T; t)$. The calculated dependences of the dielectric permittivity versus the frequency and temperature agree qualitatively with the experimental data.

Simulating dielectric spectra for electrolytes and ionic liquids

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Computer simulations provide an effective tool to access the “external” dielectric susceptibility through the calculation of microscopic correlation functions. The calculation of the screened susceptibility (i.e., the dielectric response) from simulation data is a well-established procedure only for dipolar, homogeneous systems [1]. A general treatment for charged or confined systems has so far not been found. On the one hand, present approaches for charged systems are based on the arbitrary separation between translational and rotational contributions to the susceptibility [2]. On the other hand, fluctuation formulas that link the external susceptibility to the dielectric response are so far available for a restricted set of geometries/boundary conditions [1,3] only.

We present a framework in which the actual problems and limitations are eliminated, by employing only current correlations in the calculation of dielectric properties of charged systems and, by providing a fluctuation formula for the (nonlocal [4,5]) dielectric tensor, valid in the most general case that can be addressed with classical molecular dynamics simulations. Practical examples of the importance of the ionic contribution to the dielectric response and, of the determination of the general nonlocal dielectric tensor, are provided.

Molecular dynamics in nanometer thin polymer layers

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Broadband Dielectric Spectroscopy, Spectroscopic vis-Ellipsometry, X-Ray Reflectometry, Alternating and Differential Scanning Calorimetry are combined to study glassy dynamics and the glass transition in nanometric thin (≥5 nm) layers of polystyrene (PS) having widely varying molecular weights and Polymethylmethacrylate (PMMA) deposited on different substrates. For the dielectric measurements two sample geometries are employed, the common technique using evaporated electrodes and a recently developed approach taking advantage of nanostructures as spacers. All applied methods deliver the concurring result that deviations from glassy dynamics and from the glass transition of the bulk never exceed margins of ±3 K independent of the layer thickness, the molecular weight of the polymer under study and the underlying substrate. Our findings are discussed in the context of the highly controversial literature and prove that an appropriate sample preparation is of paramount importance in order to avoid artefacts.

Heterogeneity of polymer dynamics near the glass temperature as probed by defocused imaging

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Recent efforts by single molecule defocused wide-field fluorescence microscopy (SMDWM) and complementary techniques (dielectric spectroscopy, rheology) are reviewed that monitor the 3D-reorientation of single molecules in a thin polymer film (~300 nm) near the glass transition temperature ($T_g$). The rotational correlation times obtained from SMDWM show a similar temperature dependence as the ones measured with dielectric spectroscopy and rheology, on the same polymer sample. Molecular reorientation is coupled to the segmental rather than terminal relaxation of the surrounding polymer matrix. SMDWM revealed that spatial heterogeneity is more pronounced than temporal heterogeneity within the measurement time-scale (hours to days), whereas this information is hidden in the bulk measurement.
Origin of the deviation from bulk behavior in polymers confined at the nanoscale

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A large number of experimental works highlighted how properties of ultrathin polymer films (<200 nm) depend on the annealing conditions used during their preparation (for a review, see [1]). We show clear evidence that the changes ultrathin films undergo during annealing are strongly correlated to the amount of chains irreversibly adsorbed at the interface [2]. A careful analysis of the time evolution of the dielectric function during annealing steps above $T_g$ revealed three different regimes: at short times (in comparison to the adsorption time) the thickness of the adsorbed layer is constant and the incomplete interface mimics the effect of a free surface (packing frustration); upon increase of surface coverage, the films undergo a series of metastable states characterized by the largest changes in the deviations from bulk behavior; finally the system approach a new equilibrium whose properties are fixed by the new interfacial configurations. Our picture is confirmed by further investigation of the distribution of glass transition temperatures [3], tracer diffusivity [4] and dielectric relaxation strength [5] at different distances from the adsorbing interface. Measurements of the local dielectric properties were achieved by selectively placing layers of dye-labeled polystyrene at different depth inside films of neat polystyrene.

We show that during annealing ultrathin films evolve towards a steady state whose properties might still differ from bulk. In particular, for polystyrene, the lack of specific interactions with the substrate results in slow adsorption kinetics, keeping the deviations from bulk behavior in steady states with an extremely long lifetime. Finally, we verified that the temperature dependence of the dielectric function in proximity of an interface matches the prediction of the two-order-parameter model.

Charge transport and glassy dynamics in several classes of ionic liquids confined in nanoporous silica membranes with mean pore diameters ranging between 4 nm and 15 nm are investigated in a wide frequency and temperature range by a combination of Broadband Dielectric Spectroscopy (BDS), Fourier Transform Infrared spectroscopy (FTIR), and Pulsed Field Gradient Nuclear Magnetic Resonance (PFG NMR) [1-4]. Remarkable enhancement of the ionic mobility by more than two orders of magnitude is traced down to changes in molecular conformation of the ions as a result of density reductions accompanying two-dimensional confinement. In addition, it becomes possible to determine the resulting change in density of ionic liquids in nanopores from dielectric spectra in quantitative agreement with recent molecular dynamics simulations. For some ionic liquids, slower ionic mobility is observed. This trend is fully reversed upon silanization as proved by FTIR spectra, proving the significant role of guest molecule-host membrane interaction. Technological implications of the observed confinement effects on transport properties of ionic liquids will be discussed.

References
Poster session I

Thursday, 22 September 2011

P2. C. Iacob et al., Molecular dynamics in confined imidazole liquids.
P5. K. Wegner et al., Crystallization behavior of amphiphilic PBA/PEO miktoarm star polymers.
P6. M. J. Sanchis et al., Dielectric properties of poly(2-ethoxyethyl methacrylate) membranes.
P7. M. J. Sanchis et al., Comparative study of the dipolar relaxation processes and ionic transport in two methyl succinate compounds.
P8. A. Greenbaum (Gutina) et al., The state of water in confinement near hydrophilic surfaces below the freezing temperature.
Molecular dynamics of MEO₂MA hydrogels – influence of network morphology.

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Polymer hydrogels, especially stimuli-responsive materials are one of the most significant systems for many applications (in pharmacy, medicine and many others). They are mostly synthesized from polymers exhibiting lower critical solution temperature (LCST), what in a case of crosslinked macromolecules is manifested by a volume phase transition (VPT). The interest of such materials arises from their unique properties such as ability to absorb large amount of water, high flexibility, nontoxicity, often biocompatibility, biodegradability and first of all ability to change in their properties as a consequence of even small modification in environmental conditions (temperature, pH, electrical, mechanical or magnetic fields and many others).

For many potential applications too slow response to stimuli is one of the most important problems [1]. A good solution for this issue seems to be grafting additional, dangling polymer chains exhibiting LCST on the polymer network, what should be manifested by faster response on local level and in a consequence VPT of whole hydrogel should be also faster [2].

The aim of this work is to investigate the influence of chemical structure of the polymer network, temperature, crosslinking and swelling degrees on molecular dynamics of thermo-responsive hydrogels synthesized on base of 2-(2-methoxyethoxy)ethyl methacrylate (MEO₂MA). Broadband Dielectric Spectroscopy (including high frequency region 1MHz ÷ 3GHz) was used to determine the molecular relaxation processes in the hydrogels synthesized in two ways: by Atom Transfer Radical Polymerization and by Free Radical Polymerization. Differences between hydrogels with and without dangling chains were also investigated. Additionally dry linear poly(MEO₂MA) as well as its aqueous solutions with various polymer concentration were investigated, what makes possible to identify the relaxation processes visible in analogue hydrogel samples.

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Molecular dynamics in confined imidazole liquids

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Broadband Dielectric Spectroscopy (BDS), differential scanning calorimetry (DSC) and Pulsed Field Gradient - Nuclear Magnetic Resonance (PFG - NMR) are combined to study molecular dynamics in a homologous series of imidazole-based liquids with systematic variation of the alkyl chain length. We present studies of diffusion in bulk state and in mesoporous silica membranes. By applying the Einstein-Smoluchowski relations, diffusion coefficients are obtained from BDS in quantitative agreement with independent PFG NMR measurements [1-5]. Structural α-relaxation rates and diffusion coefficients at lower temperatures are enhanced by more than two decades in 4 nm pores compared to their bulk values. This may be attributed to changes in the molecular packing due to geometrical constraints [6].

Glassy dynamics of isolated polymer molecules

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The glassy dynamics of randomly distributed, isolated poly(2-vinylpyridine) molecules is studied by means of broadband dielectric spectroscopy. Therefore, a recently developed nano-structured electrode arrangement applies which enables the investigation of samples without a full coverage of the electrode. Detailed checks of the surface topology by atomic force microscopy were performed to verify stability of the samples during the whole measurement. The examined dynamics is compared to the bulk to trace changes due to the impact of the interface and to examine the length scale of interfacial interactions.
Molecular motions in PBA/PEO miktoarm star polymers

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Miktoarm star polymers are star-shaped polymers with various types of polymer arms attached to a central core. The possibility to introduce the different functional groups into the star polymers in conjunction with the ability to self-organisation of the amphiphilic miktoarm polymers results that these materials are potential candidates for drug delivery and biological applications [1]. The goal of this study is to determine the influence of composition of poly(butyl acrylate) / poly (ethylene oxide) miktoarm star polymers on their molecular dynamics using dielectric spectroscopy and Nuclear Magnetic Resonance methods. The investigated samples with various fractions of poly(ethylene oxide) (PEO) and poly(butyl acrylate) (PBA) arms were obtained by using Atom Transfer Radical Polymerization method [2]. The analysis of temperature dependence and dispersion of NMR relaxation times as well as dielectric spectra of PBA/PEO miktoarm polymers allowed us to detect and characterize molecular motions such as: segmental and local motions of polymer chains. For all samples single glass transition process was observed. As it turned out the VFT parameters of the segmental motions in studied miktoarm polymers have similar values i.e. Vogel temperature of 167 K and fragility parameter D of 15. The investigated systems are characterized by very broad distribution of relaxation times. Moreover, the dielectric spectra show broadening of the distribution of relaxation times with increasing PEO content.

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Miktoarm star polymers represent a special type of star shape polymers, containing two or more arm species with different chemical compositions and/or molecular weights attached to a central core. Their physico-chemical properties can be modified by selection of the composition of the arms, which makes these materials suitable for many applications in such areas as catalysis, optical imaging, and medicine (as drug delivery systems) [1]. The materials under study are miktoarm star polymers consisting of various molar fractions of poly(butyl acrylate) (PBA) and poly(ethylene oxide) (PEO) arms e.g. 46/54 and 16/84. The samples were synthesized using the ATRP method by Gao at al. [2].

The goal of these investigations is to study crystallization kinetics of PBA/PEO miktoarm star polymers using differential scanning calorimetry (DSC), broadband dielectric spectroscopy (BDS) and Polarized Optical Microscopy (POM). The DSC data allow us to obtain the activation energies of crystallization process ($E_a$) having the values of 93 kJ/mol and 114 kJ/mol for the miktoarm samples with 54% and 84% PEO arms content, respectively. These values are lower than the activation energy of 161 kJ/mol for neat linear PEO (determined by Cohen et al. [3]). Lowering of the activation energy with an increase in PBA content may be explained as result of the plasticization effect, which is associated with the presence of PBA chains in the sample, which makes easier the transportation of PEO chains to the growing crystal surface during the crystallization process. Isothermal crystallization kinetics of miktoarm star polymers was monitored by dielectric spectroscopy. These data were analysed using Avrami approach and the results indicate that crystallization process occurs primary via homonucleation of PEO chains.

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Dielectric properties of poly(2-ethoxyethyl methacrylate) membranes

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The dielectric relaxation behavior of poly-2-ethoxyethyl methacrylate (PEEMA) has been studied using broadband dielectric spectroscopy (DRS) in the frequency domain of 10^{-2} to 10^8 Hz and at temperatures between 123 and 473 K. The influence of the composition, presence/absence of the crosslinking agent, on the dielectric properties of the polymers is described [1-2, 6].

The loss permittivity spectra of the membranes studied shows the existence of various relaxations processes in the temperature-frequency range studied. At high temperature and low frequencies, the isotherms representing the dielectric loss as a function of frequency show important conductive contribution overlapping the loss factor. The alpha process, associated with the dynamic glass transition and the secondary relaxations (beta and gamma processes) associated with the side-chain rotations of the asymmetric side groups, are resolved from the dielectric spectra by modelling with Havriliak-Negami (HN) function [3-4].

The alpha process corresponds to the cooperative relaxation correlated to the glass transition and arises from main-chain cooperative micro-Brownian motions and follows Vogel-Fulcher-Tammann (VFT) behavior [5]. On the other hand, the secondary relaxations follow an Arrhenius (ARR) behavior. According with our results, the local beta process originates from side-chain rotation of the asymmetric side group about the C-C bond connecting it to the polymer backbone. In the lower temperature range, these copolymers show one more relaxation, the gamma relaxation, which can be attributed to the response of the ethoxy group in the side chain.

Comparative study of the dipolar relaxation processes and ionic transport in two methyl succinate compounds

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The objective of this work was the study of the influence of slight modifications in the chemical structure of liquids on the molecular dynamics of these compounds. Electrical perturbation fields covering frequency and temperature windows of $10^{-2} – 10^6$ Hz and -130 K - 303 K were used and the models studied were 2-dicyclohexylmethyl-2-methyl succinate and dicyclohexyl-2-methyl succinate. The closeness of the chemical structure of the two methyl succinate compounds used in this work allows the fulfillment of this objective.

The spectra show the presence of several dipolar relaxations that were characterized by means of the Havriliak-Negami empirical model [1]. The results show that the temperature dependence of the dielectric strengths, relaxation times and shape parameters of the secondary and glass-liquid relaxations are very sensitive to the structure of the models.

On the other hand, traces of water and other impurities strongly affect the components of the complex permittivity of real systems at low frequencies. However, since most dielectric studies are focused on the dipolar relaxation behavior, the changes observed in the real and loss components of the complex permittivity in the low frequency region are in most cases ignored. In this work special attention is paid to the contribution of the ionic transport to the dielectric responses of the systems to electric perturbation fields in the low frequencies region where the contribution of dipolar processes to the permittivity is negligible. Special attention is paid to the temperature dependence of low frequency relaxations produced by the motion of a macrodipole arising from charges located near the liquid-electrodes boundaries [2-4].

The state of water in confinement near hydrophilic surfaces below the freezing temperature

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The main goal of the research is to find a relationship between the dynamic and the structural properties of water in hydrated heterogeneous systems. The results of dielectric spectroscopy studies of hydrated matrixes of porous glasses, clays and hydrated powders of Lysozyme are presented in wide frequency and temperature intervals. It is shown that for all studied systems the low temperature relaxation process demonstrates Arrhenius kinetics and exhibits a Cole-Cole (CC) behavior. A new phenomenological approach has been recently presented [1] that clarifies the physical mechanism of the dipole-matrix interaction in complex systems underlying the CC behaviour. Comparison with porous glasses and clays helps to understand the specific adsorbed water dynamics due to the variety in the distribution of hydration centers.

Poster session II

Friday, 23 September 2011


P.2. M. Jasiurkowska et al., Brodband dielectric and infrared spectroscopy studies on confined liquid crystals.

P.3. J. Suchanicz et al., Dielectric, thermal expansion, and heat capacity behaviour of (1-x)Na0.5Bi0.5TiO3-xBaTiO3 single crystals (x=0, 0.025, 0.0325 and 0.05).


P.5. B. Vanroy et al., Impact of finite size effects and interfacial interactions on the cold crystallization of poly(ethylene terephthalate).

P.6. V. V. Meriakri et al., Some applications of the millimeter wave spectroscopy.

P.7. B. M. Garin et al., Dielectric properties and applications at millimeter and terahertz ranges of the CVD diamonds of different types.


P.9. A. Soualmia et al., Dielectric spectroscopy and response analysis by time domain reflectometry (TDR).
Segmental and chain dynamics in thin layers of cis-polyisoprene

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Broadband Dielectric Spectroscopy (BDS) – in combination with a nanostructured electrode arrangement [1] – is used to study thin layers of cis-1,4-polyisoprene. Being a Type A polymer [2], cis-polyisoprene enables the investigation of two distinct relaxation modes taking place at two different length scales: the segmental motion which involves structures of about one nanometer in size (2 to 3 monomer units) and the normal mode which represents the dynamics of the whole macromolecule. Previous studies [3,4] involved the evaporation of a metal counter-electrode onto the spin-cast layer, the consequence of which is a so-called confinement-induced mode showing up between the normal and segmental modes for thin samples. In the current study – where highly insulating silica nanostructures are used as spacers – one interface is free. Down to 7 nm, for three molecular weights (44.5, 53 & 75 kg/mol), it is observed that: (i) the segmental mode as a local relaxation process is unaffected by the 1-D confinement; (ii) the normal mode becomes faster with decreasing layer thickness, and (iii) the so-called confinement-induced mode does not show up (Fig. 1).

Brodband dielectric and infrared spectroscopy studies on confined liquid crystals

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Broadband dielectric and Infrared spectroscopy are combined to study the molecular dynamics of the liquid crystalline compounds belong isothiocyanatobiphenyl homologous series [1,2] (abbreviated as nBT) confined in pores of diameters from 4 nm to 10.5 nm. In bulk, the studied substances show only one liquid crystalline phase: the SmE phase with orthorhombic arrangement within the molecular layers. In contrast to well-known bulk dielectric properties of nBTs, confinement leads to modification of the molecular dynamics. Two relaxation processes are detected. The slower process corresponds to molecular reorientation around short axis and it is faster in pores than in bulk. The second process is attributed to a librational motion of the molecules close to the walls. Both processes exhibit an Arrhenius-type temperature dependence. The crystallization temperature is significantly reduced in comparison to the value for bulk and it depends on pore sizes. Detailed analysis of the temperature dependent infrared spectra indicates the different impact of confinement on the rigid and flexible molecular units of nBTs.

Dielectric, thermal expansion and heat capacity behaviour of 
(1-x)Na_{0.5}Bi_{0.5}TiO_3-xBaTiO_3 single crystals 
(x=0, 0.025, 0.0325 and 0.05)

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Lead-free (1-x)Na_{0.5}Bi_{0.5}TiO_3-xBaTiO_3 single crystals (x=0, 0.025, 0.0325 and 0.05) were prepared by Czochralski method. The obtained samples show pure perovskite structure with rhombohedral symmetry at room temperature. The dielectric, thermal expansion and heat capacity measurements of these crystals have been made in a wide temperature range. The broad anomalies of thermal expansion and heat capacity, which approximately correspond to a structural, ferroelectric and dielectric properties anomaly, were observed. It was concluded that these anomalies can be connected with the temperature features of polar regions and with the formation of long-range ferroelectric phase. The Burns temperature was determined and was found to increase with increasing BaTiO_3 content. The local polarization was calculated from the thermal expansion data. Low frequency (100Hz-100kHz) investigations reveal the diffuse phase transitions. The obtained results are discussed in terms of local electric and strain fields caused by the difference between the ionic radii of (Na,Bi) and Ba ions. The NBT-BT system is expected to be a new promising candidate for lead-free electronic crystals.
Log-periodic corrections to the Cole-Cole law

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Using the previously developed model based on self-similar (fractal) structure of a substance on mesoscale region [1, 2] new method of derivation of the kinetic equations describing evolution of the total polarization and containing the integro-differential operators of non-integer order has been developed. The solution of these equations in frequency domain leads to the generalized Cole-Cole empirical law for the complex dielectric permeability. It has been proved (with proper mathematical accuracy) that the power-law exponent $\alpha$ determining the Cole-Cole law coincides with dimension of the space-temporal fractal ensemble. In supposition of discrete scale invariance of the space-temporal structure of the fractal medium considered the new expression for the complex dielectric permeability has been obtained. It contains the log-periodic corrections and, hence, it generalizes the conventional Cole-Cole expression

$$
\varepsilon(\omega) = \varepsilon_\infty + \frac{\varepsilon_a - \varepsilon_\infty}{1 + R(i\omega)} - \sum_{k=-\infty}^{\infty} C_k (d_f)(i\omega \tau_0)^{-d_f+i/2k}
$$

The new generalized Cole-Cole law can contain the multi-peak structure in behavior of the imaginary part and multi-step structure in behavior of the real part of the complex permeability, correspondingly. As it is well-known that multi-peak dielectric spectra are trying to describe usually by means of linear combination of the H-N functions. But this empirical and naive approach contains one essential drawback because for these additive combinations of empirical functions (where each term is associated with one relaxation process) it is impossible to write the common kinetic equation for the total polarization describing its behavior in the temporal region. But if the basic hypothesis related to self-similar structure of the disordered medium considered is correct then new generalized Cole-Cole function can replace easily the previous empirical hypothesis based on the linear combination of the H-N (or other) functions. This essential replacement contains new possibilities in analysis of a wide class of the disordered (self-similar) media because any fitting parameter entering into new expression has a clear physical meaning and tightly related with micro/meso-scopic processes taking place in the substance considered.

Impact of finite size effects and interfacial interactions on the cold crystallization of poly(ethylene terephthalate)

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We investigated the impact of nanometric confinement on the cold crystallization of ultrathin films of poly (ethylene terephthalate). Upon reduction of the thickness, in agreement with previous investigations of other polymers [1,2], we observed an increase of the cold crystallization temperature during heating scans and of the characteristic crystallization time, during isothermal experiments. To discriminate between interfacial and size effects we performed experiments on different substrates. While in the lower crystallization rate in thicker films was imputed to nucleation effects, a thickness threshold of ~30 nm indicating the onset of interfacial interactions could be identified. Contrary to the general idea on the impact of interfaces on the dynamics of confined systems, we verified that the interfacial energy is not a reliable parameter to predict the deviations from bulk behavior.

Some applications of the millimeter wave spectroscopy

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Applications for liquids. 1.1. The method [1], developed for determining small differences in the complex permittivity of lossy dielectrics, is applied to measure the dielectric properties of water solutions with small content of glucose, as well as of human skin, in the millimeter wave (MMW) range. The results of these measurements are used to implement a real-time, including noninvasive, technique for determining small glucose content in water and blood. In particular, a clear correlation between the glucose content and the reflection coefficient from skin after an oral glucose tolerance test on an empty stomach is established. 1.2. It is shown that MMWs allow one to carry out real-time nondestructive control of blends of biodiesel (BD) with conventional mineral diesel (MD). The highest sensitivity to the composition of blends is achieved when the electromagnetic-wave absorption is measured in the short MMW band. In blends with 80% of MD and 0–20% of BD, this method allows one to determine the content of BD to within 1% without temperature correction. 1.3. It is found that the measurement of the absorption of 90–100-GHz MMWs in palm and soya oils in the temperature interval of 130–190 °C allows one to detect a difference between fresh oil and oil used in a cooking process. This difference can be used for non-destructive on-line monitoring of deterioration of oil during cooking. 1.4. Two methods, based on a waveguide with a longitudinal slot [2] and on a dielectric waveguide, are elaborated for determining the content of alcohol in water.

Applications for solid materials. 2.1. The dielectric properties of a wide variety of building and common use materials are measured, which are of great interest for wave communication systems, introscopy, imaging and monitoring, and desinfection of culture objects. 2.2. A new method [3] for determining the complex refractive index of thin lossy films (semiconductors, carbon nanotubes) on low-loss substrates is developed. This method consists in measuring the reflection coefficients on either side of the structure formed by a given substrate with a thin lossy film and an auxiliary dielectric matching plate. 2.3. Application of the MMW dielectric spectroscopy to control the concentration and uniformity of the distribution of nanoparticles in polymers, for example, fire retardant nanoparticles and carbon nanotubes in some polymers (Nylon 6, PLA) and water in Metal-Organic Frameworks.

Acknowledgment. The authors are sincerely grateful to S. Bourbigot, W. Fang, P. Hildeman, K.L. Lu, and T.M. Wu for supplying experimental samples. This work was supported in part by the RFBR projects nos. 10-0213310 RT_OMI and 11-02-93965.

Dielectric properties and applications at millimeter and terahertz ranges of the CVD diamonds of different types

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Conventionally, high quality polycrystalline diamond films are prepared by using microwave plasma chemical vapor deposition (MPCVD) technique [1,2]. But on the other hand, the deposition rate of the MPCVD diamonds is rather low. Last time, a high power direct current arc plasma jet (APJ) technique has been developed, which shows the advantages of higher deposition rate and lower material cost [3]. The dielectric loss at broad frequency and temperature ranges ($f = 30$–$400$ GHz and $T = 30$–$900$ K) of the MPCVD and APJ diamonds are described and compared. The loss mechanisms are discussed.

In MPCVD diamonds the loss tangent at millimeter (MM) range decreases with frequency that is due to electric conductance in intergranular regions [1,2]. On the contrary, the loss in APJ diamond increases with frequency. It corresponds to the mechanism of loss induced by lattice disorder [4]. Also, the concentration of defects with shallow levels in the forbidden gap on the order of magnitude is revealed compared MPCVD diamonds by the charge-based deep level transient spectroscopy (Q-DLTS) measurements.

The refractive index APJ diamond is essentially less compared with usual MPCVD diamonds. It means that the density of sample is less. It combines with absence of big internal cavities in the APJ diamond. The latest can give additional advantage at terahertz (THz) range due to less scattering of electromagnetic wave.

The present MPCVD diamonds are characterised by record breaking low losses and acceptable for megawatt gyrotron windows at continuous wave operation (up to at least 2 MW) at MM range and for application in the Int. Thermonuclear Experimental Reactor (ITER) project. And a fundamental opportunity is for essentially more powers is follows from the comparison with theory [1]. The APJ diamonds are perspective for diamond coated copper-diamond composites in the high power electronics at MM and THz ranges.

Dielectric spectroscopy and thermogravimetric analysis study of 2-hydroxyethylacrylate-co-2-hydroxyethylmethacrylate hydrogels

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Hydrogels have been used in many pharmaceutical and biomedical applications, i.e. drug delivery systems, contact lenses and wound dressings. The aim of this contribution is to study the physicochemical properties of 2-hydroxyethylacrylate-co-2-hydroxyethylmethacrylate (HEA-HEMA) hydrogels depending on the copolymer composition using thermogravimetric analysis (TGA) and broadband dielectric spectroscopy (BDS).

The hydrogel samples were synthesised by three-dimensional free-radical copolymerisation of HEA and HEMA [1]. Eleven hydrogel samples with different HEA / HEMA ratios were prepared starting from Poly-HEMA (HEA0:HEMA100) to Poly-HEA (HEA100:HEMA0) with 10 mol % step in HEA-HEMA composition. TGA (PerkinElmer) measurements were performed in temperature range from 25°C to 200°C at 5°C/min. BDS (Novocontrol Alpha system) spectra were recorded in frequency range from 0.1Hz to 1MHz and temperature range from -140°C to 80°C.

TGA experiment has shown that water is evaporated from hydrogels in two steps: at low temperatures the loss of free water occurs and at high temperatures the loss of bound water (Fig. 1). The total amount of water as well as of the bound water dramatically increases for HEA-rich samples (> 30 mol %), whereas the percentage of free water remains independent on the hydrogel composition.

Dielectric spectroscopy measurements revealed different behavior in HEMA-rich and HEA-rich samples (Fig. 2). One relaxation process with the activation energy about 20 kJ/mol was registered for samples with HEA content greater than 50 mol %, whereas two relaxation processes (with ~20KJ/m and ~43KJ/m) can be distinguished for HEMA-rich samples. The low frequency process is attributed to the relaxation of hexagonal ice and the high frequency process is related to mobility of the ice strongly defected by polymeric chains [2].

Dielectric spectroscopy and response analysis by time domain reflectometry (TDR)

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Equipe Spectroscopie Diélectrique, Laboratoire de Physique des Matériaux, Faculté de Physique USTHB Alger

It is known that dielectric characterization[1] presents a fundamental and applied aspect the goal is to determine basic parameters that are found in several empirical laws. Based on these parameters, Debye equation seems the most appropriate especially for polar and slightly conductive material as for example water [2] and some alcohols.

In fact, all the empirical laws are designed to bring the process of dielectric losses by conductivity due to migration of charges and dielectric losses by relaxation due to the reorientation of dipoles in an electromagnetic field. In this paper, we propose an equivalent circuit model to describe in good agreement the two processes.

The typical experimental set up consists of the TDR miniaturized unit [3], the processing control software, the coaxial transmission line, the coaxial cell filled with the material and termination load of admittance 0S or of impedance 50 Ohms.

The propagated step voltage along the transmission line meets an impedance break, causing the TDR signal to be reflected; the reflected signal carries the signature of the device under test (DUT) [4].

The objective of this paper is to present an approach of models describing the polarization mechanisms including dielectric relaxation and electrical conductivity [5].

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6. J. Colmenero (Spain), 16
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8. I. Ermolina (UK), 50
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10. G. Floudas (Greece), 30
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13. A. Greenbaum (Israel), 41
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27. R. R. Nigmatullin (Russia), 24, 46
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33. A. Schönhals (Germany), 26, 34
34. M. Sega (Germany), 28
35. A. Serghei (France), 18, 36, 43
36. A. Sokolov (USA), 12
37. A. Soualmia (Alger), 51
38. J. Suchanicz (Poland), 45
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45. K. Wegner (Poland), 38
46. M. Wübhenhorst (Belgium), 15, 31, 48
The ModuLab MTS is a new concept in testing the electrical properties of materials. The system uniquely combines time domain tests (I-V, pulse, ramp), and AC tests (C-V, Mott-Schottky, impedance, permittivity), together with a wide range of modules providing high voltage, high current resolution, sample / reference measurements. Temperature control and sample holder accessories add to the capabilities of the system.

<table>
<thead>
<tr>
<th>Polymers</th>
<th>Ceramics</th>
<th>Dielectrics</th>
</tr>
</thead>
<tbody>
<tr>
<td>MTS provides a wide range of capabilities for the electrical and temperature testing of polymers that are used in many applications - cable insulation, substrates for displays, low-k dielectrics for semiconductors, conducting polymers, plastic coatings, etc.</td>
<td>Ceramics are often used in high temperature and insulator applications - aerospace turbine blades, space shuttle tiles, engine nozzles, sensors, disk brakes, etc. MTS provides high voltage, high temperature, and low current options that are ideal for testing ceramic materials.</td>
<td>Ferroelectrics (used in electronic applications e.g. PC memory), ceramics, polymers, oils, glass and porcelain are all examples of dielectric materials (non-conductors of electricity). Testing often requires high voltage, excellent current resolution and high accuracy.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Semiconductors</th>
<th>Solar Cells</th>
<th>Displays</th>
</tr>
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<tbody>
<tr>
<td>Semiconductors materials change resistivity depending on an applied electric field, with applications in PDs, rectifiers, solar cells, amplifiers etc. A wide range of AC and time domain test techniques are needed, including I-V, C-V, temperature control, and wide voltage / current range.</td>
<td>Solar cells make use of the inexhaustible free energy supply from the sun as an alternative to fossil fuels. MTS provides I-V characterization (to evaluate power / efficiency) and Impedance / C-V to determine charge carrier density and mobility, all in one unit - reducing cost and complexity.</td>
<td>New display technologies such as OLED and AMOLED are enabling great advances in portable computers, cell phones and thin screen TVs. A wide range of test techniques are employed including pulse (for display hold / flicker tests), I-V, C-V and impedance.</td>
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<table>
<thead>
<tr>
<th>Nanomaterials</th>
<th>Biomaterials</th>
<th>Superconductors</th>
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<tbody>
<tr>
<td>Nanomaterials are being developed that combine existing ceramics, polymers etc., with nanomaterials to produce composites with amazing mechanical, thermal and electrical properties. The versatility and modularity of ModuLab MTS is key for testing these new materials.</td>
<td>ModuLab MTS is able to test a wide range of biomaterials using time domain and AC techniques - viability of transplant organs, medical implant materials, blood, virus or tissue cells, and for testing drug delivery systems and pharmaceutical products.</td>
<td>Superconductors are materials that have zero resistance below a critical temperature. They are used in electromagnets, MRI, NMR and mass spectrometers. The ability to measure extremely low impedance while accurately controlling temperature is essential for testing these materials.</td>
</tr>
</tbody>
</table>

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Broadband Dielectric Spectroscopy and its Advanced Technological Applications
Modulab MTS software is a very flexible and comprehensive materials test software package. A large selection of test types are provided, from standard open circuit, I-V, pulse, C-V, mith Schottky to complete multi-step sequences that can include sample temperature control using a cryostat or high temperature furnace.

As test parameters are entered into the software, a waveform diagram displays the timing and levels that will be applied to the cell when the test is run.

Equivalent circuit models may be constructed using a range of components including resistors, capacitors, inductors, distributed elements, constant phase elements, Ganscher elements, and Warsburg open / short elements.

A built-in report generator takes test results and outputs them, together with graphs, diagrams and analysis information into your selected word processor software.

Ordering information (hardware)

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<tr>
<th>Module</th>
<th>Description</th>
</tr>
</thead>
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<td>Time domain analyzer (pulse, I-V)</td>
</tr>
<tr>
<td>MREF (2096A)</td>
<td>Sample / reference dielectric option</td>
</tr>
<tr>
<td>MBST 2A (2096/2A)</td>
<td>2A high current booster option</td>
</tr>
<tr>
<td>MHV 100 (2096/1A)</td>
<td>100 V high voltage option</td>
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<tr>
<td>MTA (2096/3A)</td>
<td>Femto ammeter low current option</td>
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<td>MFAA 1MHz (2066B)</td>
<td>1 MHz FRA option (for AC tests)</td>
</tr>
<tr>
<td>Chas 06 (2100A)</td>
<td>8 slot chassis</td>
</tr>
<tr>
<td>Chas 04 (2101A)</td>
<td>4 slot chassis</td>
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</table>

Ordering information (software)

<table>
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<tr>
<th>Module</th>
<th>Description</th>
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<tbody>
<tr>
<td>Modulab MTS</td>
<td>Software included with the system controls chassis, options and temperature accessories</td>
</tr>
</tbody>
</table>

Optional accessories

<table>
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<th>Module</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>129610A</td>
<td>5K - 600K Cryostat with solid / liquid sample holder</td>
</tr>
<tr>
<td>129602A, 60A, 64A</td>
<td>Solid / liquid sample holder</td>
</tr>
<tr>
<td>TBA</td>
<td>High temperature furnace with sample holder</td>
</tr>
<tr>
<td>TBA</td>
<td>Ultra high voltage amplifier</td>
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